

SGS North America Inc.
Standard Operating Procedure

**Standard Operating Procedure for PCCD/Fs & PCBs in Water
by Mega-Talex**

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Revision History

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1.0 Scope and Application

- 1.1 To describe the procedures followed for the extraction and fractionation of PCDD/Fs and PCBs in 10-20 L of water (drinking and effluent) using temperature-assisted liquid-liquid extraction with an appropriate organic solvent.
- 1.2 See Document DC_139 Appendix D for a full list of definitions.

2.0 Summary of Method

- 2.1 Liquid samples such as drinking, ground water and wastewater samples can be extracted using the TALEX technique. A measured volume of sample is extracted with ~15 mL of toluene (PCDD/Fs) or ~15 mL isooctane (PCBs or PCDD/F-PCB combo) while boiling the water for 120 minutes. The water vapors carrying the PCDD/F and PCB congeners condense inside the column. The condensed water is then forced to pass through a layer of toluene or isooctane before returning to the boiling flask. The analytes partition into the organic layer. The resulting extract is dried through sodium sulfate, concentrated, fractionated and analyzed by HRGC-HRMS.

3.0 Safety Precautions

- 3.1 Gloves must be worn when handling samples, standards, and reagents.
- 3.2 Safety glasses must be worn at all times when working in the laboratory.
- 3.3 A lab coat must be worn while dealing with samples, standards, and reagents.
- 3.4 In the interest of safety and pollution prevention, all spills must be cleaned up immediately.

4.0 Interferences and Preventive Measures

- 4.1 Solvents, reagents, incorrectly cleaned glassware, and other extraction techniques may yield interference components in the final extract.
- 4.2 Care should be taken in cleaning any extraction vessel or equipment that the sample or sample extract may contact during the extraction process.
- 4.3 Interferences co-extracted from samples vary from matrix to matrix and from sample to sample. Additional cleanup may be required as needed or specified by project.
- 4.4 Contamination or carry-over may occur by extraction of high level samples sequentially with clean or low level samples. Proper record

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keeping allows for review of how and in what order samples were extracted if a high level sample is discovered during analysis. This makes it possible to determine which samples may have carry over and need re-extraction.

5.0 Sample Collection, Preservation and Storage

Not applicable.

6.0 Equipment and Supplies

- 6.1 Appropriately sized round bottom
- 6.2 Custom Talex column
- 6.3 Boiling stones
- 6.4 Chiller
- 6.5 Balance
- 6.6 .45 µm filter paper
- 6.7 Graduated cylinder
- 6.8 Heating mantle
- 6.9 Appropriate micropipettes
- 6.10 pH strips
- 6.11 Residual chlorine test strips
- 6.12 Glass beads
- 6.13 Glass wool
- 6.14 Fume hood
- 6.15 Rotovap
- 6.16 Mini acid column

7.0 Standards and Reagents

- 7.1 Cleaned DI water

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- 7.2 Sodium sulfate
- 7.3 Concentrated sulfuric acid
- 7.4 Hexane
- 7.5 Toluene
- 7.6 Isooctane
- 7.7 Extraction standards
- 7.8 Tetradecane
- 7.9 Clean-up standards
- 7.10 Injection standards

8.0 Calibration

- 8.1 Pipette Calibration
 - 8.1.1 Refer to SGS document MI_46 for pipette and syringe calibration procedure.

9.0 Sample Preparation

- 9.1 Aqueous Samples
 - 9.1.1 Use the custom-designed Mega-TALEX column.
 - 9.1.2 Prime the column with a few mL (usually ~15 mL) of DI water.
 - 9.1.3 Add 15 mL of toluene for PCDD/Fs or 15 mL isooctane for (PCDD/Fs &) PCBs.
 - 9.1.4 Add the demister in the mid section of the column (glass beads) and top the column with a secondary demister (glass wool).
 - 9.1.5 Transfer the water sample to the 20-L (or larger) round bottom flask. Measure and record the volume, 1 L (or 2 L) or less, before or after the transfer using a graduated cylinder.
 - 9.1.6 Measure pH and residual chlorine using test strips and record in the paperwork.
 - 9.1.7 Adjust the pH to ~5-7 with concentrated sulfuric acid if necessary.
 - 9.1.8 Fortify the aqueous samples with the extraction/analytes standards directly into the round bottom.
 - 9.1.9 Add several boiling stones (Do not use Carborandum). Make sure there are enough glass beads sitting at the bottom of the flask. Floating chips do not regulate boiling.

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- 9.1.10 Allow chiller water to circulate through the condenser making sure to check for leaks and /or cracks. Do not use water with a temperature higher than 15°C.
- 9.1.11 Start heating by turning the knob to position “70” on the thermo controllers.
- 9.1.12 Record the time from the moment the water starts boiling. Allow the boiling to go on for 120 minutes.
- 9.1.13 After 120 min, turn off the heat and allow the system to cool down for approximately 30 minutes.
- 9.1.14 Slowly drain the water layer to waste.
- 9.1.15 Drain the toluene or isooctane through a sodium sulfate plug prepared with the glasswool that was used at the top of the column directly inside a collection vessel containing 20 µL of tetradecane for PCDD/F or into a 100-mL round bottom flask for PCBs.
- 9.1.16 If necessary, tip the column to draw out most of the organic solvent.
- 9.1.17 Rinse the inside of the column with 3x3 mL of toluene for PCDD/Fs (or hexane for (PCDD/Fs &)PCBs); draining directly through the sodium sulfate plug and collecting the rinses with the original extract.
- 9.1.18 Add the CS as specified on the paper work.
- 9.1.19 For PCDD/Fs, concentrate to near dryness inside the vacuum oven or in the fume hood to where there are no traces of toluene left. For PCBs, rotovap down to the isooctane level.
- 9.1.20 If the sample is a clean water sample (e.g., drinking water), add the JS and transfer inside a GC/MS vial.
- 9.1.21 For other samples, subject the residue to ASECS or a mini Acid column. In such case, add 0.25 mL of tetradecane before the solvent exchange step for PCDD/Fs, or load the isooctane extract for the PCBs..

10.0 Analytical Procedure

Not applicable.

11.0 Details of Calculations

Not applicable.

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12.0 Quality Control Requirements

Not applicable.

13.0 Data Review and Reporting Requirements

Not applicable

14.0 Preventative Maintenance

Not applicable

15.0 Tables

Not applicable

16.0 Definitions

- 16.1 DI water – deionized water
- 16.2 PCDD/F - polychlorinated dibenzo-p-dioxin/furan
- 16.3 PCB - Polychlorinated Biphenyls
- 16.4 HRGC – High resolution gas chromatograph
- 16.5 HRMS – High resolution mass spectrometer
- 16.6 P&P – Pulp and paper
- 16.7 ES – Extraction standard
- 16.8 CS – Clean-up standard
- 16.9 JS – Injection standard

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- 16.10 ASECS – Clean-up unit
- 16.11 SPE – Solid phase extraction
- 16.12 SPE-C – Solid phase extraction with carbon
- 16.13 COC – Chain of custody

17.0 References

- 17.1 SW-846 Method 3520C Revision 3, December 1996
- 17.2 SGS document DC_139, Appendix D Definitions.
- 17.3 SGS document MI_278, Waste Disposal and Pollution Prevention.
- 17.4 SGS document MI_46, Pipette and Syringe Calibration.
- 17.5 SGS document MI_141, Review of Analytical Data.
- 17.6 SGS document DC_358, PCCD/Fs in water by SPE